

6,6,8a-Trimethyl-3a,6,7,8a-tetrahydrobenzo[*b*]furo[3,2-*d*]furan-2,4(3*H*,5*H*)-dione

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Key indicators

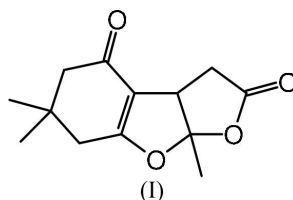
Single-crystal X-ray study
 $T = 120\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.027
 wR factor = 0.068
 Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The structure of the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_4$, comprises a non-planar chiral molecule where the cyclohexene double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å).

Comment

The title compound, (I), a perhydrofurobenzofuran, exhibits hypoglycemic properties. A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) for related structures reveals that there are 38 compounds containing a six-membered carbocyclic ring with two linked five-membered furo rings, as in (I). However, in all 38 molecules the C_6 ring is benzene; none are cyclohexane, -ene or -yne variants. The structure of (I) comprises a non-planar chiral molecule where the $\text{C5}=\text{C10}$ double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å). The two torsion angles that highlight the non-planarity of the molecule are $\text{O1}-\text{C1}-\text{C4}-\text{C3}$ [$-127.2(1)^\circ$] and $\text{O2}-\text{C1}-\text{C4}-\text{C5}$ [$104.0(1)^\circ$].



Experimental

The title compound was prepared according to the literature procedure of Nagarajan *et al.* (1988). Crystals were grown from ethanol.

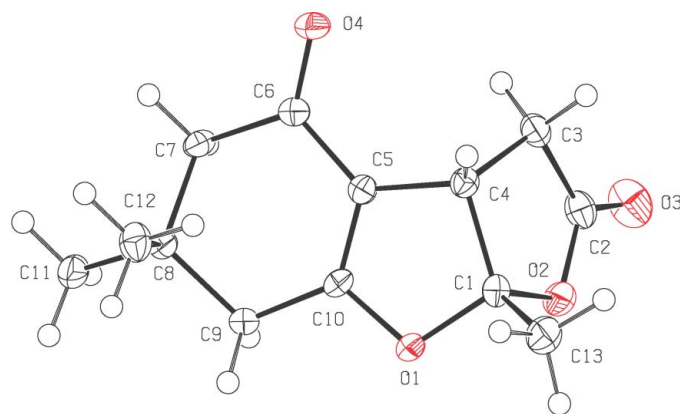


Figure 1
 The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

Crystal data

$C_{13}H_{16}O_4$
 $M_r = 236.26$
 Orthorhombic, $P2_12_12_1$
 $a = 9.4853$ (3) Å
 $b = 10.2904$ (2) Å
 $c = 12.2872$ (4) Å
 $V = 1199.32$ (6) Å³
 $Z = 4$
 $D_x = 1.309$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 1560 reflections
 $\theta = 2.9$ – 27.5°
 $\mu = 0.10$ mm⁻¹
 $T = 120$ (2) K
 Prism, colourless
 $0.50 \times 0.40 \times 0.40$ mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.953$, $T_{\max} = 0.962$
 8608 measured reflections
 2343 independent reflections

2235 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 26.0^\circ$
 $h = -11 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.068$
 $S = 1.04$
 2343 reflections
 158 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.2043P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.14$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.045 (6)

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.98 (CH₃), 0.99 (CH₂) and 1.00 Å (CH). The isotropic displacement

parameters for all H atoms were set equal to $1.25U_{\text{eq}}$ of the carrier atom. In the absence of significant anomalous scattering effects, the 763 Friedel pairs were merged.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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supporting information

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C₁₃H₁₆O₄
 $M_r = 236.26$
 Orthorhombic, $P2_12_12_1$
 Hall symbol: P 2ac 2ab
 $a = 9.4853$ (3) Å
 $b = 10.2904$ (2) Å
 $c = 12.2872$ (4) Å
 $V = 1199.32$ (6) Å³
 $Z = 4$
 $F(000) = 504$

$D_x = 1.309$ Mg m⁻³
 Melting point: 434 K
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 Cell parameters from 1560 reflections
 $\theta = 2.9$ – 27.5°
 $\mu = 0.10$ mm⁻¹
 $T = 120$ K
 Prism, colourless
 $0.50 \times 0.40 \times 0.40$ mm

Data collection

Nonius KappaCCD
 diffractometer
 Radiation source: Bruker Nonius FR591
 rotating anode
 10 cm confocal mirrors monochromator
 Detector resolution: 9.091 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2003)

$T_{\min} = 0.953$, $T_{\max} = 0.962$
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 2343 independent reflections
 2235 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.9^\circ$
 $h = -11 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.068$
 $S = 1.04$
 2343 reflections
 158 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.2043P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.14$ e Å⁻³
 Extinction correction: SHELXL97,
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.045 (6)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.884134.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.16781 (9)	0.17367 (8)	−0.01057 (7)	0.0201 (2)
O2	0.10135 (9)	0.36301 (8)	0.07462 (7)	0.0225 (2)
O3	0.03946 (10)	0.44551 (10)	0.23535 (9)	0.0360 (3)
O4	0.46337 (9)	0.08915 (8)	0.27867 (7)	0.0235 (2)
C1	0.21985 (13)	0.29930 (11)	0.02444 (10)	0.0184 (3)
C2	0.12818 (14)	0.39484 (12)	0.18079 (11)	0.0228 (3)
C3	0.27705 (13)	0.36048 (12)	0.20951 (10)	0.0216 (3)
H31	0.2807	0.3129	0.2795	0.027*
H32	0.3358	0.4397	0.2155	0.027*
C4	0.32810 (12)	0.27457 (11)	0.11621 (10)	0.0172 (3)
H4	0.4266	0.2959	0.0934	0.021*
C5	0.30737 (12)	0.13117 (11)	0.13465 (10)	0.0162 (2)
C6	0.37348 (12)	0.04866 (12)	0.21468 (10)	0.0167 (3)
C7	0.32064 (13)	−0.09032 (12)	0.21643 (10)	0.0184 (3)
H71	0.2375	−0.0952	0.2649	0.023*
H72	0.3950	−0.1463	0.2480	0.023*
C8	0.27957 (13)	−0.14482 (11)	0.10420 (10)	0.0175 (3)
C9	0.17135 (13)	−0.05357 (11)	0.04923 (10)	0.0180 (3)
H91	0.1627	−0.0758	−0.0289	0.022*
H92	0.0778	−0.0648	0.0836	0.022*
C10	0.21803 (12)	0.08331 (11)	0.06087 (9)	0.0163 (3)
C11	0.21468 (14)	−0.27965 (12)	0.11981 (12)	0.0252 (3)
H111	0.1899	−0.3162	0.0487	0.032*
H112	0.1297	−0.2725	0.1647	0.032*
H113	0.2830	−0.3366	0.1560	0.032*
C12	0.41107 (14)	−0.15744 (12)	0.03283 (11)	0.0255 (3)
H121	0.3847	−0.1938	−0.0380	0.032*
H122	0.4792	−0.2150	0.0684	0.032*
H123	0.4535	−0.0715	0.0224	0.032*
C13	0.26637 (16)	0.37458 (13)	−0.07371 (11)	0.0273 (3)
H131	0.1876	0.3821	−0.1249	0.034*
H132	0.3449	0.3293	−0.1090	0.034*
H133	0.2970	0.4615	−0.0514	0.034*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0239 (5)	0.0165 (4)	0.0198 (5)	0.0007 (3)	−0.0071 (4)	0.0029 (3)
O2	0.0182 (4)	0.0236 (4)	0.0258 (5)	0.0048 (4)	−0.0010 (4)	−0.0006 (4)
O3	0.0276 (5)	0.0399 (6)	0.0405 (6)	0.0016 (5)	0.0124 (5)	−0.0100 (5)

O4	0.0222 (5)	0.0247 (5)	0.0235 (5)	−0.0021 (4)	−0.0091 (4)	0.0003 (4)
C1	0.0190 (6)	0.0159 (5)	0.0203 (6)	0.0016 (5)	0.0010 (5)	−0.0002 (5)
C2	0.0232 (7)	0.0187 (6)	0.0264 (7)	−0.0034 (5)	0.0041 (5)	−0.0013 (5)
C3	0.0253 (6)	0.0178 (6)	0.0217 (6)	−0.0012 (5)	−0.0001 (5)	−0.0029 (5)
C4	0.0165 (6)	0.0164 (6)	0.0186 (6)	−0.0005 (4)	0.0003 (5)	0.0018 (5)
C5	0.0151 (6)	0.0167 (5)	0.0167 (5)	0.0007 (4)	0.0008 (5)	−0.0001 (4)
C6	0.0144 (6)	0.0203 (6)	0.0154 (6)	0.0006 (4)	0.0009 (5)	−0.0008 (5)
C7	0.0166 (6)	0.0204 (6)	0.0183 (6)	−0.0008 (5)	−0.0040 (5)	0.0044 (5)
C8	0.0171 (6)	0.0159 (5)	0.0195 (6)	0.0003 (5)	−0.0037 (5)	0.0015 (5)
C9	0.0191 (6)	0.0180 (6)	0.0169 (6)	0.0001 (5)	−0.0037 (5)	−0.0005 (5)
C10	0.0144 (5)	0.0186 (6)	0.0158 (6)	0.0030 (5)	−0.0006 (5)	0.0026 (4)
C11	0.0247 (6)	0.0182 (6)	0.0327 (7)	−0.0016 (5)	−0.0080 (6)	0.0033 (5)
C12	0.0237 (7)	0.0222 (6)	0.0306 (7)	0.0038 (5)	0.0035 (5)	−0.0025 (5)
C13	0.0358 (7)	0.0242 (7)	0.0220 (6)	−0.0015 (5)	0.0007 (6)	0.0057 (5)

Geometric parameters (Å, °)

O1—C10	1.3646 (14)	C7—H71	0.99
O1—C1	1.4492 (14)	C7—H72	0.99
O2—C2	1.3689 (16)	C8—C12	1.5303 (17)
O2—C1	1.4399 (15)	C8—C11	1.5300 (16)
O3—C2	1.1956 (16)	C8—C9	1.5464 (16)
O4—C6	1.2324 (15)	C9—C10	1.4835 (17)
C1—C13	1.4997 (17)	C9—H91	0.99
C1—C4	1.5461 (17)	C9—H92	0.99
C2—C3	1.4978 (18)	C11—H111	0.98
C3—C4	1.5265 (16)	C11—H112	0.98
C3—H31	0.99	C11—H113	0.98
C3—H32	0.99	C12—H121	0.98
C4—C5	1.5059 (16)	C12—H122	0.98
C4—H4	1.00	C12—H123	0.98
C5—C10	1.3351 (17)	C13—H131	0.98
C5—C6	1.4426 (16)	C13—H132	0.98
C6—C7	1.5156 (16)	C13—H133	0.98
C7—C8	1.5388 (17)		
C10—O1—C1	107.34 (9)	H71—C7—H72	107.6
C2—O2—C1	111.83 (10)	C12—C8—C11	108.83 (10)
O2—C1—O1	105.50 (9)	C12—C8—C7	109.77 (10)
O2—C1—C13	109.81 (10)	C11—C8—C7	108.66 (10)
O1—C1—C13	108.80 (10)	C12—C8—C9	110.02 (10)
O2—C1—C4	106.33 (9)	C11—C8—C9	109.78 (10)
O1—C1—C4	107.21 (9)	C7—C8—C9	109.76 (9)
C13—C1—C4	118.43 (11)	C10—C9—C8	109.65 (10)
O3—C2—O2	120.52 (12)	C10—C9—H91	109.7
O3—C2—C3	129.37 (12)	C8—C9—H91	109.7
O2—C2—C3	110.08 (11)	C10—C9—H92	109.7
C2—C3—C4	105.00 (10)	C8—C9—H92	109.7

C2—C3—H31	110.7	H91—C9—H92	108.2
C4—C3—H31	110.7	C5—C10—O1	114.02 (10)
C2—C3—H32	110.7	C5—C10—C9	127.24 (11)
C4—C3—H32	110.7	O1—C10—C9	118.74 (10)
H31—C3—H32	108.8	C8—C11—H111	109.5
C5—C4—C3	114.40 (10)	C8—C11—H112	109.5
C5—C4—C1	100.62 (9)	H111—C11—H112	109.5
C3—C4—C1	103.99 (9)	C8—C11—H113	109.5
C5—C4—H4	112.3	H111—C11—H113	109.5
C3—C4—H4	112.3	H112—C11—H113	109.5
C1—C4—H4	112.3	C8—C12—H121	109.5
C10—C5—C6	121.44 (11)	C8—C12—H122	109.5
C10—C5—C4	110.01 (10)	H121—C12—H122	109.5
C6—C5—C4	128.51 (11)	C8—C12—H123	109.5
O4—C6—C5	122.45 (11)	H121—C12—H123	109.5
O4—C6—C7	122.60 (11)	H122—C12—H123	109.5
C5—C6—C7	114.92 (10)	C1—C13—H131	109.5
C6—C7—C8	114.51 (10)	C1—C13—H132	109.5
C6—C7—H71	108.6	H131—C13—H132	109.5
C8—C7—H71	108.6	C1—C13—H133	109.5
C6—C7—H72	108.6	H131—C13—H133	109.5
C8—C7—H72	108.6	H132—C13—H133	109.5
C2—O2—C1—O1	120.97 (10)	C1—C4—C5—C6	−176.76 (11)
C2—O2—C1—C13	−121.94 (11)	C10—C5—C6—O4	174.96 (11)
C2—O2—C1—C4	7.32 (12)	C4—C5—C6—O4	−2.42 (19)
C10—O1—C1—O2	−104.33 (10)	C10—C5—C6—C7	−6.91 (16)
C10—O1—C1—C13	137.90 (10)	C4—C5—C6—C7	175.71 (11)
C10—O1—C1—C4	8.71 (12)	O4—C6—C7—C8	−147.51 (11)
C1—O2—C2—O3	−178.12 (11)	C5—C6—C7—C8	34.36 (14)
C1—O2—C2—C3	3.55 (14)	C6—C7—C8—C12	66.75 (13)
O3—C2—C3—C4	168.91 (13)	C6—C7—C8—C11	−174.33 (10)
O2—C2—C3—C4	−12.95 (13)	C6—C7—C8—C9	−54.28 (13)
C2—C3—C4—C5	−92.39 (12)	C12—C8—C9—C10	−75.43 (12)
C2—C3—C4—C1	16.37 (12)	C11—C8—C9—C10	164.83 (10)
O2—C1—C4—C5	103.98 (10)	C7—C8—C9—C10	45.45 (13)
O1—C1—C4—C5	−8.49 (11)	C6—C5—C10—O1	−178.35 (10)
C13—C1—C4—C5	−131.95 (11)	C4—C5—C10—O1	−0.53 (14)
O2—C1—C4—C3	−14.69 (11)	C6—C5—C10—C9	1.03 (19)
O1—C1—C4—C3	−127.17 (10)	C4—C5—C10—C9	178.85 (11)
C13—C1—C4—C3	109.37 (12)	C1—O1—C10—C5	−5.33 (13)
C3—C4—C5—C10	116.43 (11)	C1—O1—C10—C9	175.23 (10)
C1—C4—C5—C10	5.63 (13)	C8—C9—C10—C5	−21.53 (17)
C3—C4—C5—C6	−65.96 (16)	C8—C9—C10—O1	157.83 (10)